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AUGER MEASUREMENTS ON T-027 SAMPLES EXPOSED DURING THE SKYLAB 2 MISSION

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TECHNICAL MEMORANDUM X-64834

AUGER MEASUREMENTS ON T-027 SAMPLES EXPOSED DURING THE SKYLAB 2 MISSION

INTRODUCTION

The contamination of surfaces exposed to space environmental conditions has posed the possibility of serious degradation of sensitive surfaces such as optical elements, windows, and thermal control devices. The vacuum environment and solar radiation effects enhance the outgassing of materials, the evaporation of fairly high vapor pressure materials, and the transport from sources to sensitive surfaces. These effects, coupled with zero-g properties, create conditions and problems requiring a broad range of investigation, not only into the properties of materials used but also into the mechanisms involved in these various processes. Investigations of contaminant sources, contaminant transport, contaminating mechanisms involving adsorption and chemical modifications, and the removal of contaminants are being conducted by various groups. The Auger measurements described herein represent one contribution to these activities. The goals of this investigation were to examine surfaces exposed to several directions in space relative to Skylab, to identify any semipermanent contaminants adsorbed in terms of their atomic species, to obtain relative quantities, and to observe any obvious anisotropy in contaminant distribution.

Because many precautions have been exercised in the selection of materials for space applications based on considerable ground-based research, the levels of contamination have been significantly reduced; however, the unique, high pumping speeds in space, the high probability that nonequilibrium conditions exist, and numerous other conditions make it desirable to continue to investigate contamination and its control. Because of the large number of parameters involved, numerous experimental measurements are needed. Quartz crystal microbalances can provide quantitative information in the form of adsorbed mass changes, ellipsometry and reflectance measurements can provide data on optical changes and film thicknesses, and numerous other measurements can sample the vapor phase, etc. Auger electron spectroscopy (AES) allows semiquantitative identification of atomic species with high sensitivity. As with all measurements, AES suffers certain limitations, which will be described later. The most significant limitation for the present space measurements is the requirement that the contamination be of a semipermanent type so that measurements

can be made with the present ground-based system. To emphasize this point, two main categories of contaminants will be described, those which are transient and exist only as long as the sources remain active (e.g., fogging of a bathroom mirror) and those which are permanent. Obviously, the transient type requires in-situ studies, and any ground-based investigation must rely on reasonably permanent species, especially if the measurement must be done in vacuo, as is the case for AES measurements.

Usually materials which readily desorb or evaporate from the sources will also readily desorb from surfaces which they contaminate slightly beyond a monolayer; therefore, the high vapor pressure materials should decay as their sources decay. Factors that could alter this situation are (1) source temperatures that are higher than the temperatures of the sensitive surfaces because of different orientations relative to the sun and (2) chemical changes. such as polymerization, occurring after adsorption on the sensitive surfaces. The transport of low vapor pressure materials to the sensitive surfaces also poses another problem. Since the diffusion and desorption mechanisms are exponentially dependent upon temperature and since the vacuum environment permits much larger temperature differences than could exist with convective heat transfer, surface orientations relative to sunlight are important considerations. Likewise, line of sight and subtended solid angle considerations are important, since the mean free path is large for the vacuum involved, and transport over great distances can occur. Thus, specific geometries and locations are very important considerations. The ultraviolet component of sunlight and other forms of radiation are effective stimuli for polymerization of high vapor pressure, low molecular weight species into low vapor pressure species [1,2]. Considering other possible chemical bonding processes as well, materials can thus be transformed into more permanent contaminants similar in process to photoresist or epoxy being cured. Finally, transport of particulates or droplets can occur because dumps, rocket firings, vibrations, etc., can release such material, which can travel large distances in vacuo under zero-g conditions. Particles and droplets in vacuo on earth would rapidly fall to the chamber floor with no gas to support them, while in zero g the particles will float freely or travel in orbital paths modified by whatever forces act upon them; electrostatic forces limited in range by shielding effects of the plasma environment could represent one of these forces.

As is apparent from the above discussion, each situation with its specific geometries, sources, and other parameters needs to be studied on an individual basis to determine the overall effect of contamination for each case; however, certain conclusions on the general effects of contamination in space can be drawn also.

PRINCIPLES OF AES

Auger electrons are produced when an electron is ejected from an inner shell of an atom and an outer shell electron fills the vacancy; this process is also responsible for the photons of discrete energy which are ejected as a competing process for satisfying the conservation principles. The Auger electrons are ejected from the outer shells and have discrete energies determined by the difference in energy between shells minus the energy required to escape from the atom; additional small corrections for surface work function, etc., are also applicable. The French physicist Pierre V. Auger originally produced the ionization of the atoms with X rays. The use of electron beams with several kilo electron volts of energy is more common today, and instruments with high sensitivity (capable of detecting approximately 10-3 of a surface monolayer) are commercially available. The energies of the Auger electrons are typically determined by a cylindrical mirror analyzer, as shown in Figure 1. The actual signal is processed to provide a differentiated output which enhances the weak Auger peaks that are superimposed on the secondary electron current. Thus, differentiated peaks are observed which correspond to discrete transitions associated with the particular atomic species producing them. Typical spectra of this type are shown in Figures 2 and 3, where the differentiation of each Auger peak produces the positive-zero-negative response corresponding to the change in slope of the signal as a positive peak is scanned. Numerous detailed articles on AES are available in the literature [3-5].

Because the Auger electrons are of relatively low energy and their cross sections for interaction with atoms are large, only those atoms ionized near the surface are likely to produce detectable spectra. Generally the majority of the intensity arises from the outermost monolayers. Gold peaks disappear when the gold is covered by only five monolayers of silver, and similar coverages of other materials should limit the range of detection to less than 3 nm (30 Å) below the surface [6,7]. This restricted region of measurement makes AES a valuable tool for surface studies. To investigate layers below the surface, it is possible to remove the overlying layers by argon ion bombardment. By using low-energy, low-current ion beams, it is possible to gradually remove layers from the surface. However, caution is required in this process because the sputtering yields of various materials vary and the process does disrupt the surface to some extent.

MEASUREMENT RESTRICTIONS

Since AES must be performed in a vacuum, it is not possible to study species which are readily desorbed unless cooling of the samples is used.

Hydrogen does not produce Auger electrons; therefore, the presence of hydrocarbons, a common source of contamination, must be based on the observation of carbon alone and inferences from other observations.

Although chemical information is available from the electronic spectra, as evidenced by electron spectroscopy for chemical analysis (ESCA) measurements, most AES instruments do not presently provide information readily associated with a given molecular structure. The best approach for investigating molecular species appears to be the study of infrared spectra by means of attenuated total reflectance and internal reflection spectroscopy [8-10].

AES is basically a qualitative measurement with quantitative aspects. While absolute quantities are not identified, except by comparison to standards, the relative quantities of a given atomic species on a surface are easily compared; thus, the experiment described here was designed with a mask covering 50 percent of each sample's surface to provide a step function in the concentration of anticipated contaminants produced by line of sight transport to the surfaces.

Often materials deposited with less than 5 nm (50 Å) thickness do not form continuous films but exhibit island formation; as more material is deposited, these islands may coalesce into continuous films. The finite size of the electron beam used to ionize the atoms will integrate over a sizeable area such that the substrate can produce spectra even though a fair amount of contamination may be present. This occurs if the contaminant does not cover the whole surface under the electron beam (the beam used in this study was approximately 1 mm in diameter but covered an elliptically shaped area approximately 1 mm by 3 mm since it was incident at a grazing angle). Also, step function changes in a contaminant or a contaminant located at a single point will appear to have a distribution when the surface is scanned because of this integration over the area. To an extent, corrections can be made for this averaging effect.

MEASUREMENT CONCEPTS IN RETROSPECT

Previous AES measurements had demonstrated the high sensitivity to surface contaminants. Exposure even to the cleanest environments provided detectable contamination changes. The question of most concern initially was whether or not the measurement was too sensitive. To investigate this question, initial measurements were made by cleaning samples and exposing them to laboratory air which was bled into the vacuum chamber through filters. Subsequently, samples were loaded in a special shipping container (Fig. 4) by pressurizing the vacuum chamber with dry nitrogen and handling the specimen holders with clean-room type gloves through a bag sealed to the chamber's port. This glove box addition was made by attaching clean room bagging materials to the gloves and sealing the bag except for a slit through which the pressure could escape. With this arrangement, an air current was sensed at the chamber port because of the nitrogen leaving the chamber, thus suggesting that entry of contamination would be hindered. The samples were loaded into the special container and sealed in this environment. They were then transported to a 10K clean room where the process of adding and removing masks was simulated. One sample was double-bagged (inner bag nylon) and another placed in a stainless steel shipping container. These were returned to the chamber by reversing the previous operations and they were examined for contamination.

Although the samples showed changes, they were much less contaminated than when they were originally placed in the chamber (following washing and degreasing with solvents). Thus, it was felt that cleaning to a moderately clean state by ion bombardment in the chamber and exposure to filtered laboratory air provided a satisfactory state for characterizing the cleanliness of the surfaces before flight. Any attempt to absolutely clean the surfaces (a very difficult task under the best conditions) seemed unnecessary, and it seemed that with reasonable handling precautions the samples would remain clean enough to detect significant contaminating events during exposure in orbit.

To simplify the analysis, each specimen was to have 50 percent of its surface covered by a mask so that any contamination of a permanent nature would show up as a step function when the surface was scanned along a line perpendicular to the edge of the mask. For comparison, two control samples were to be flown in the T-027 compartment (exposed to the space vacuum but not extended into space), an additional control was to be left in the shipping container, and other samples were to be left in the chamber in the laboratory. In addition, visual observations with both black light and white light were to be

utilized. If significant contamination occurred during the flight, the samples were to be gradually cleaned by ion bombardment while changes were observed with AES until the substrates were visible on each half of each sample and the atomic species was identified by the Auger spectra.

DETAILS OF SAMPLE PREPARATION

The overall dimensions of the samples were defined for the T-027 experiment as disks, 2.540 cm in diameter by 0.318 cm thick. Specimens were machined and polished to fit stainless steel holders of this size (MSFC Drawing Number 61M10009). Each holder, which will be referred to as the sample, was designed to securely hold both one gold and one nickel specimen and to provide a removable stainless steel cover which masked 50 percent of each specimen's surface (Fig. 5).

The nickel specimens were produced from high purity single crystals. and the gold specimens were vacuum-coated gold films on stainless steel. Accelerated aging of the gold films was simulated by heating test specimens in air with no adverse effects; the coating adhered very well, surviving both tape tests and scraping of the surfaces. When the specimens were assembled into the samples, it was easy to handle the samples with clean gloves without actually contacting the specimen surfaces; this was an added advantage. After the specimens were carefully machined to size, they were mounted in stainless steel jigs and ground and polished to the proper tolerances using metallographic techniques. The front surfaces were polished to mirror finishes with 0.3 and $0.05~\mu m$ aluminum oxide on felt disks. They were removed from the polishing jig and cleaned with detergent and water until all traces of polishing compound were removed, then they were repeatedly cleaned in acetone followed by alcohol and were blown dry with a filtered Freon can held upright (improperly used Freon cans have been observed to leave contaminants if liquid Freon reaches the surface). These specimens were mounted as samples and placed in the vacuum chamber in a sample holder specially designed for 10 samples (Fig. 6).

Each sample could be positioned in front of either the Auger electron analyzer or the ion bombardment gun. It was found that the samples initially exhibited very large carbon peaks, and considerable ion bombardment was required to obtain reasonably clean specimens with a fairly uniform but tolerable level of contamination. After exposure to filtered air for 3 days and subsequent chamber evacuation (an ion pump was used), the specimen surfaces

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were scanned and data were recorded. The samples were loaded into the shipping container, which was then sealed and carried to the clean room where masks were installed. Two controls were treated similarly and returned to the chamber, where scans showed modest changes, as expected. The remainder of the masked samples in the shipping container were turned over to Dr. J. A. Muscari (Principal Investigator for the T-027 experiment on Skylab 2) as guest samples. These samples were handled in 10K or better environments with gloves or clean instruments until loaded in the T-027 housing and sealed for flight.

EXPOSURE AND SUBSEQUENT ANALYSIS

As previously stated, the Auger measurements were performed in vacuo on samples transported back to earth after exposure to the orbital environment. Details of the exposure of samples in the experiment are available from Dr. Muscari. Briefly, the Auger samples were exposed for approximately 40 hours while facing four different directions (+X, -X, +Y, and -Y), where +X was toward the Command Service Module (CSM) and +Z was toward the sun. The exposure was to be accomplished by extension of the sample array toward the sun from the solar port. However, problems encountered early in the Skylab 1 mission required erection of a sun shield from this port; this modified the plans so that the array was extended from the antisolar port in a direction away from the sun (-Z direction). The samples were, thus, in the shadow of the spacecraft at all times, where the temperatures were estimated to be approximately -32° C. Thus, the magnitude of the effect of contamination due to sources at higher temperatures should be significantly less, as should any possible polymerization effects due to scattered solar radiation. The assigned positions for samples in the T-027 array and the directions they faced are as follows:

MSFC Sample	T-027 Sample Array Position	Direction in Space
043H	32	- X
043E	63	+Y
043K	80	_Y
043M	89	+X (toward CSM)
043N	211	flight control
043D	216	flight control

Also:

043 J ground control sample in shipping container

After the shipping container was received at the Marshall Space Flight Center (MSFC) following exposure and recovery, the seal was broken, and sample removal was carried out inside the vacuum chamber by using the glove box arrangement with positive nitrogen pressure in the chamber as previously described. Each sample was removed individually, the cover was removed, and the sample was placed in its location in the sample holder. After all samples were loaded in the holder, a glass window was sealed to the port through which these operations took place. Visual inspection of the samples showed no apparent changes in the flight samples; all appeared uniform and highly polished.

Examination of all samples with black light showed no detectable contamination on any of the specimens. Evacuation of the chamber was accomplished by sorption pumps to a pressure of approximately 1.0 μ m, and ion pumps were used to continue the evacuation to less than $10^{-2} \mu m$ before the Auger was turned on. Brief scans showed that all surfaces exhibited Auger peaks associated with the substrate materials, indicating no continuous contaminant films of appreciable thickness. Typical spectra for both the nickel and gold specimens before and after flight are shown in Figures 7 and 8. These spectra were taken from specimens of nickel and gold which had been sputtered reasonably clean by argon ion bombardment and then exposed to filtered laboratory air for 3 days (to simulate later handling conditions). Similar spectra were taken at intervals across each specimen to provide a profile along the diameter which was perpendicular to the mask's knife edge. Since the carbon and oxygen peaks at approximately 270 eV and 515 eV, respectively, were predominant on all specimens, their peak-to-peak heights were plotted as a function of position on the diameter. The 850 eV peak for nickel and the 146 eV peak always associated with the gold substrate were plotted for each sample in a like manner. Although there were larger peaks associated with the gold and the nickel and their peak-to-peak heights appear to change more (the lower energy electrons are less likely to escape through contaminants), the 850 eV and 146 eV peaks were arbitrarily chosen as representative of the samples. It should be noted that the relative heights of the different species do not correspond to exact ratios for the quantities of these species; choosing the largest peaks associated with the substrates would have indicated larger relative peaks for substrates initially and less after the flight, enhancing the apparent contaminating effect to some extent. It should also be pointed out that one of the gold peaks is just below the carbon peak at 270 eV and they overlap in most of the measurements, making it slightly more difficult to determine the relative amount of carbon present.

The preflight and postflight profiles are plotted in Figures 9 through 16, and the profiles from the same postflight samples after being in vacuo for 7 weeks are shown in Figures 17 and 18.

INTERPRETATION OF THE DATA

The height above the zero level on Figures 9 through 18 corresponds to the measured AES peak-peak height for the three species plotted. The relative heights measured across the surface provide an accurate profile for the distribution of each separate species; however, the ratios of the heights when comparing different species do not represent exactly the ratios of the number of atoms of these species, since the spectra were not normalized against standard spectra. Attempts were made, however, to keep all parameters of the measurements fixed so that the relative heights between postflight and preflight spectra would be representative of relative changes in each species.

The rapid changes near the ends of the profiles occur because the electron beam is overlapping and stimulating Auger spectra from the stainless steel holders which have higher impurity levels. This effect is also accompanied by the appearance of iron in the spectra. Thus, the outer boundaries of the profiles should be questioned and large changes there not taken seriously.

Species other than those plotted were seldom seen. The few exceptions were small, single peaks at highly isolated locations and were not readily identified with a definite atomic species. The sharply localized nature suggested a dust particle. On occasions when the electron beam struck the crevice between the specimens and their holders, rapidly fluctuating spectra of many lines were observed but disappeared within minutes, suggesting that trapped, fairly high vapor pressure species were released by the electron bombardment, with the outgassing ceasing rather rapidly. The changes could be observed on the oscilloscope display but were too rapid to be accurately recorded with the 30-sec sweep rate of the x-y recorder. Such outgassing species were investigated by mass spectrographic techniques prior to receiving the samples.

CONCLUSIONS

The lack of a well-defined step where the mask was located on each specimen indicates that highly directional contaminants of a permanent nature were apparently not deposited to any extent. The peaked nature of some of the

profiles, however, indicates localized concentrations. Whether these concentrations are particulate or are due to a more continuous distribution is difficult to determine; however, both forms could be present.

The fact that the substrates are visible on all specimens indicates that no continuous film exceeding 5 or 10 monolayers and of a permanent nature was deposited on the specimens. This is in agreement with the maximum value of 3 nm (30 Å) film thickness verbally reported from ellipsometry measurements by Dr. Muscari.

The generally more uniform profiles across the control samples and their somewhat lower peak heights, compared to the profiles of some of the exposed samples, suggest less exposure of the controls to contaminating effects. In any case, a low rate of permanent contamination buildup is indicated for the locations and orientations of all these samples. This does not exclude the possibility of transient species having been present at one time, however. The outgassing observed when the electron beam impinged on the crevices at some of the boundaries between specimens and holders suggests that at least token transient species were present.

Although AES provides no measure of changes in optical properties, the visual observations made under white light indicated no apparent changes, and the black light produced no observable fluorescence associated with surface contaminants. More detailed optical measurements were made by others participating in the T-027 experiments. A typical threshold of 5 nm (50 Å) contamination for degrading effects on UV optics, however, indicates that optical degradation should not have been significant on the samples.

Verbal reports have been received from Dr. Robert Naumann (MSFC) of contamination effects occurring on the solar side; if confirmed, they will emphasize the importance of location, orientation, etc., with respect to sources, solar radiation, and other parameters, some of which are described in the introduction.

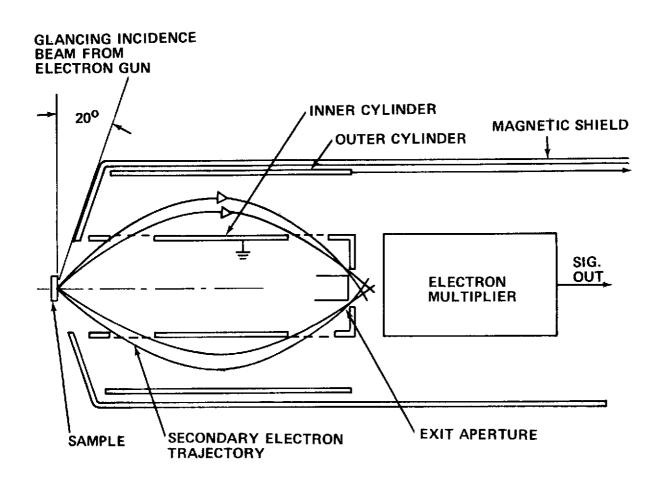


Figure 1. Schematic diagram of cylindrical mirror analyzer.

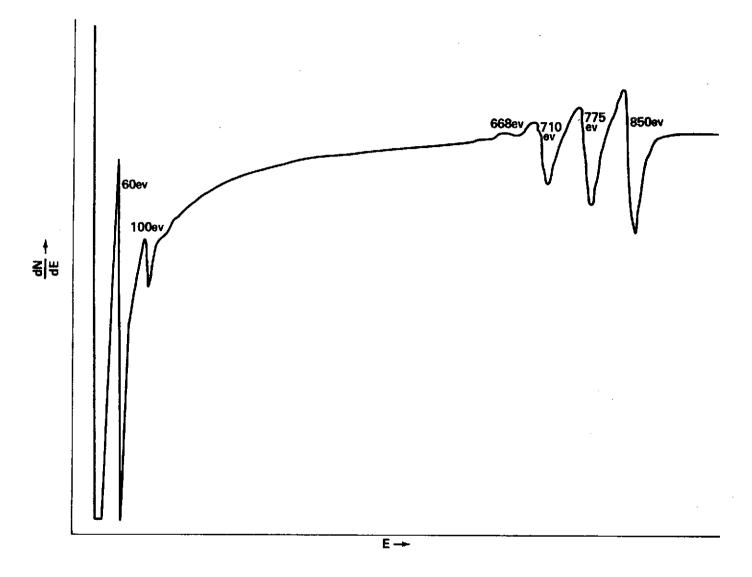


Figure 2. AES of clean nickel surface.

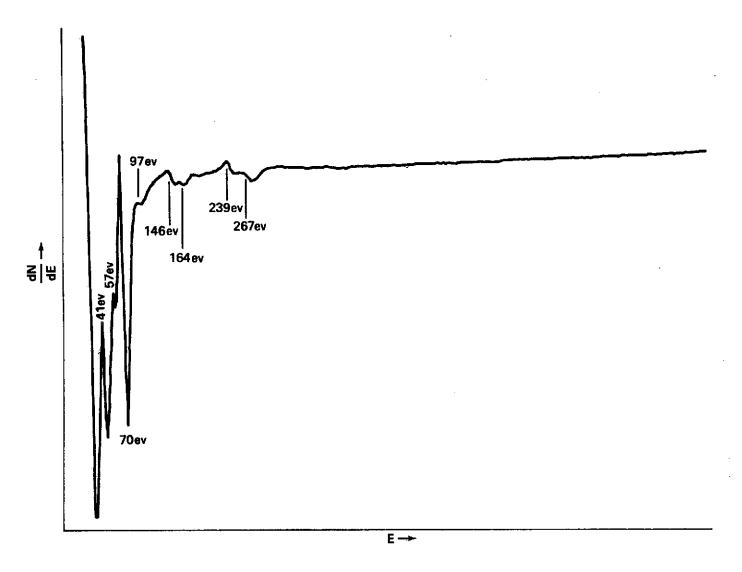


Figure 3. AES of gold specimen 043H after ion bombardment cleaning.

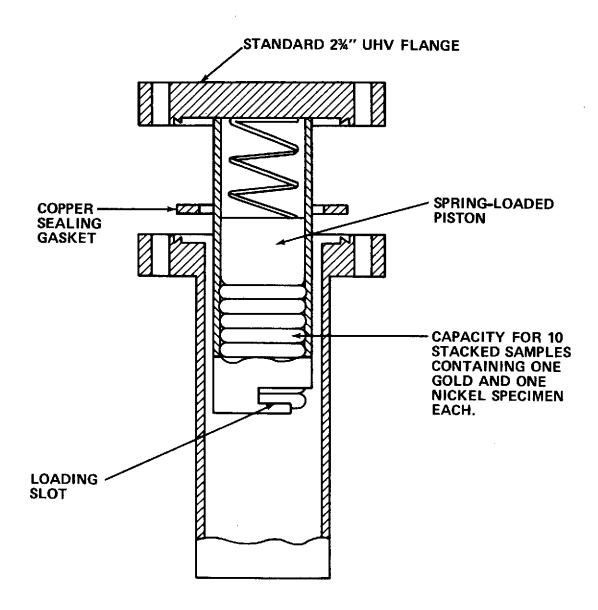


Figure 4. Stainless steel shipping container for samples.

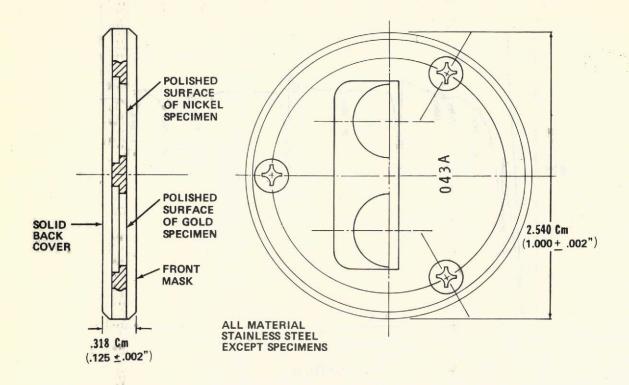


Figure 5. Sample consisting of assembly of gold and nickel specimens with cover mask.

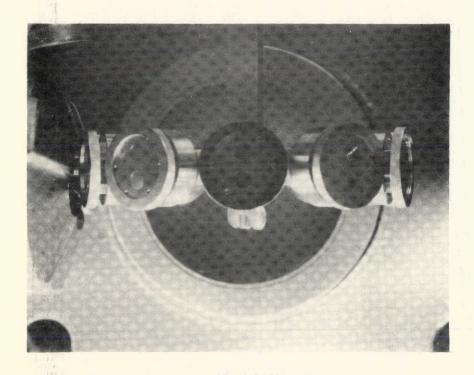
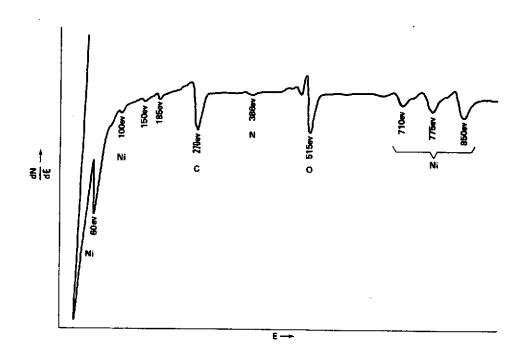
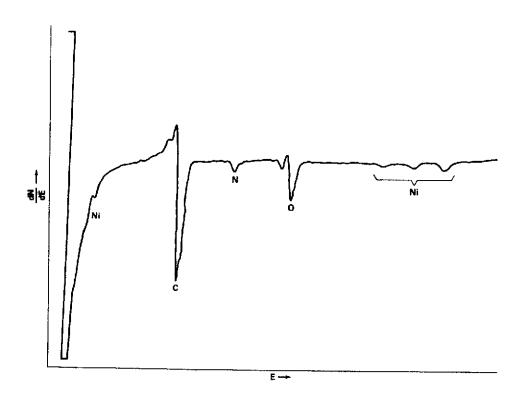


Figure 6. Photo showing 10 samples mounted for measurements with front masks removed.

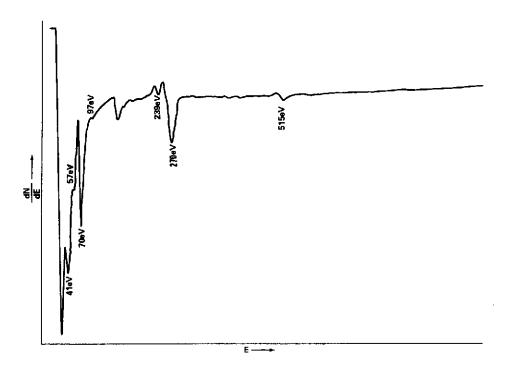


a. Preflight.

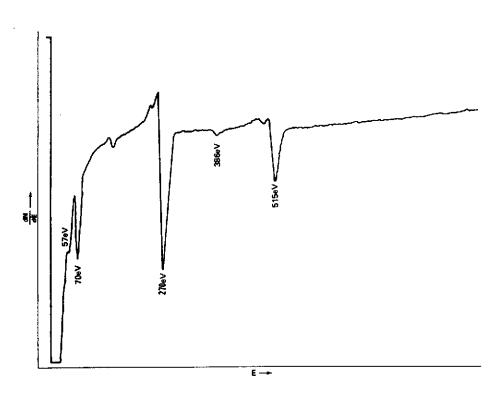


b. Postflight.

Figure 7. Example of nickel specimen.

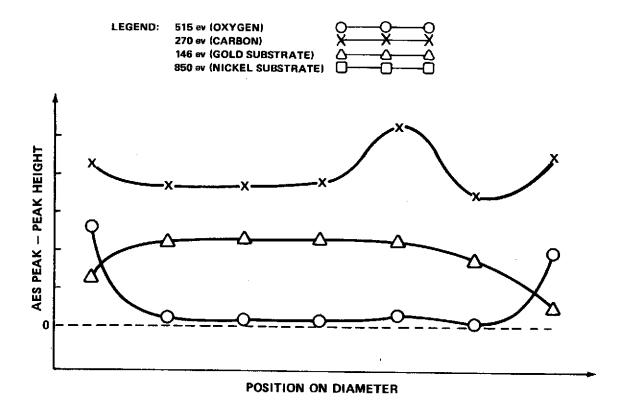


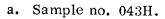
a. Preflight

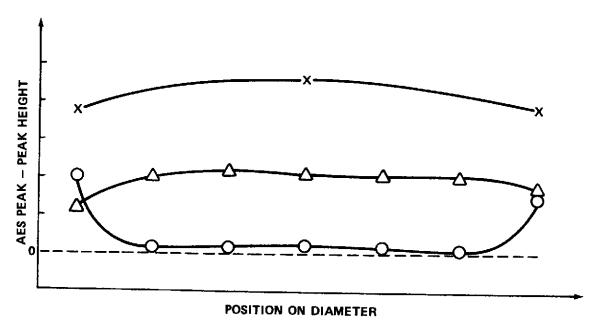


b. Postflight.

Figure 8. Example of gold specimen.

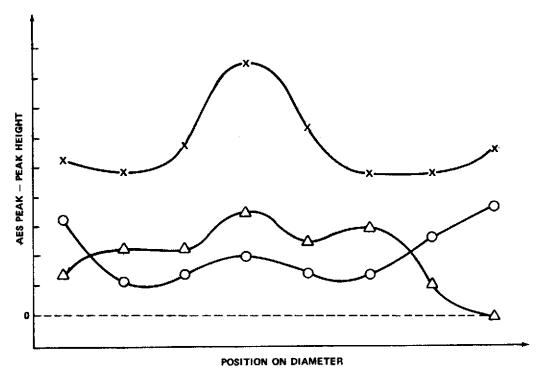




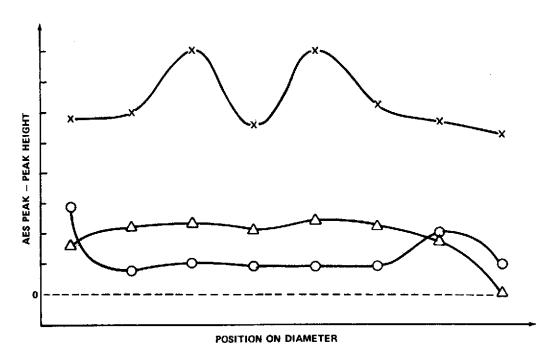


b. Sample no. 043E

Figure 9. Preflight, gold specimen exposed to filtered air.

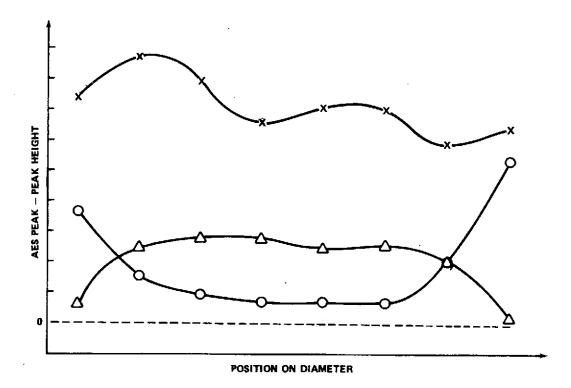


c. Sample no. 043K.

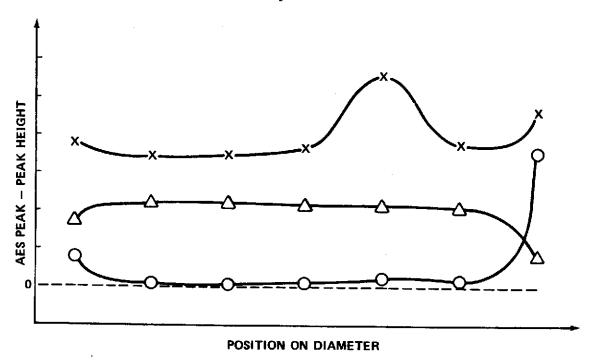


d. Sample no. 043M.

Figure 9. (Continued)

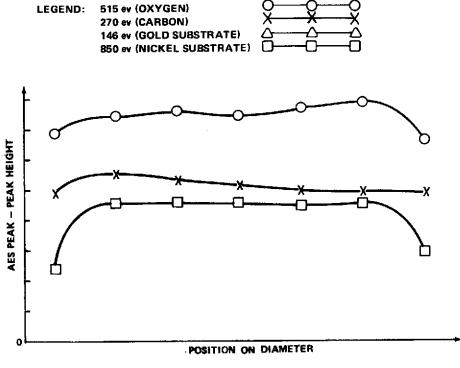


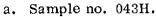
e. Sample no. 043N.

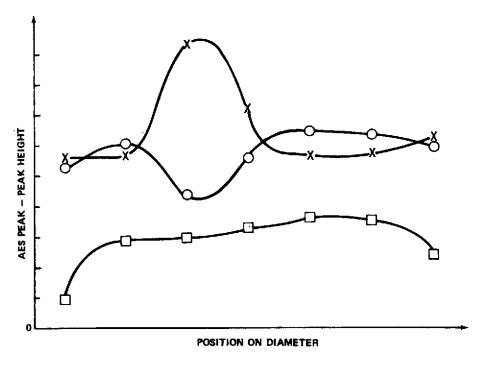


f. Sample no. 043J.

Figure 9. (Concluded).

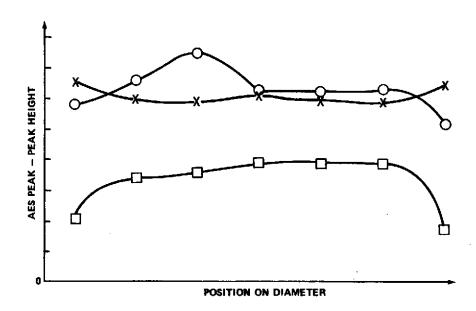




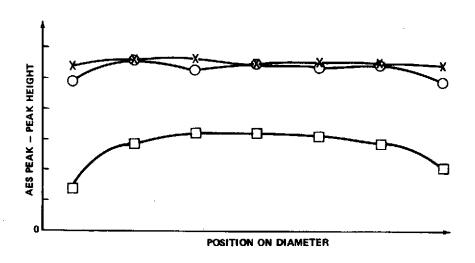


b. Sample no. 043K.

Figure 10. Preflight, nickel specimen exposed to filtered air.

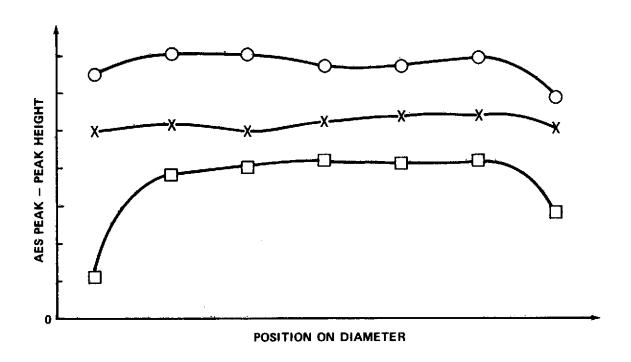


c. Sample no. 043M.



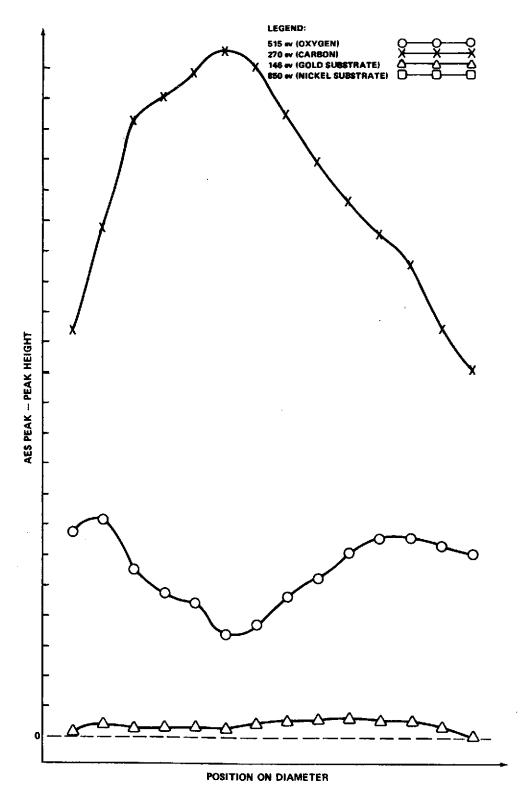
d. Sample no. 043N.

Figure 10. (Continued).



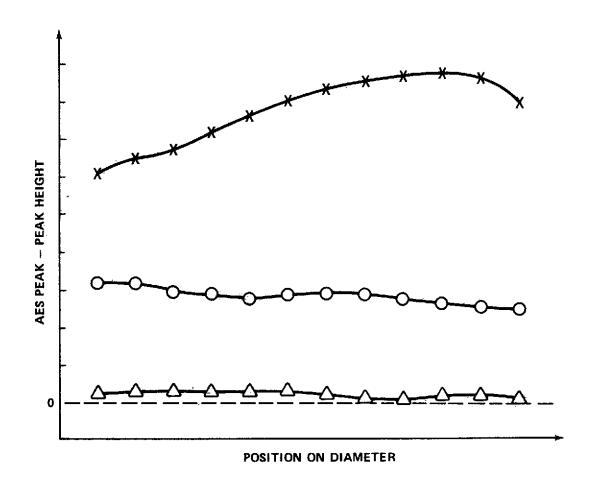
e. Sample no. 043J.

Figure 10. (Concluded).



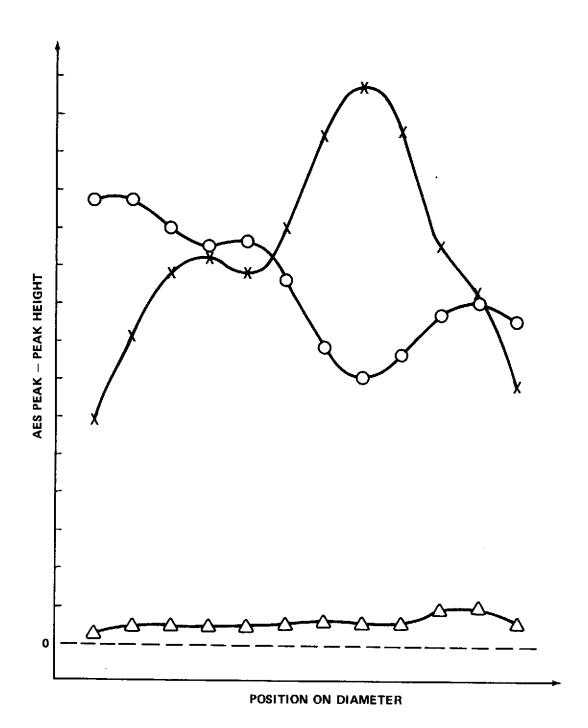
a. Sample no. 043H, -X direction.

Figure 11. Postflight, gold specimen exposed in four directions.



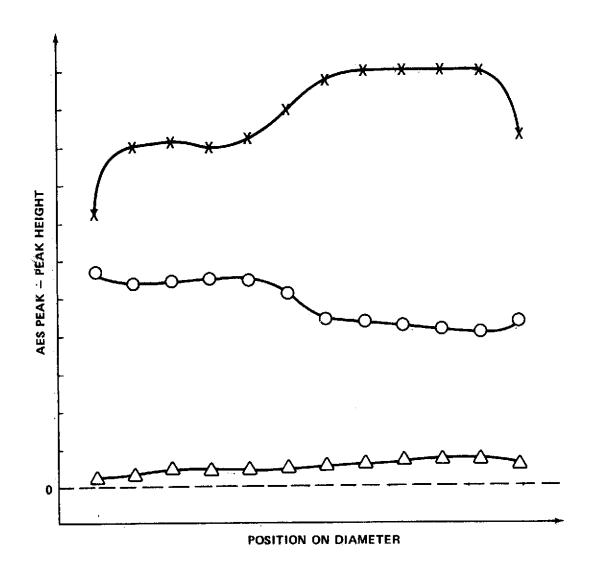
b. Sample no. 043E, +Y direction.

Figure 11. (Continued).



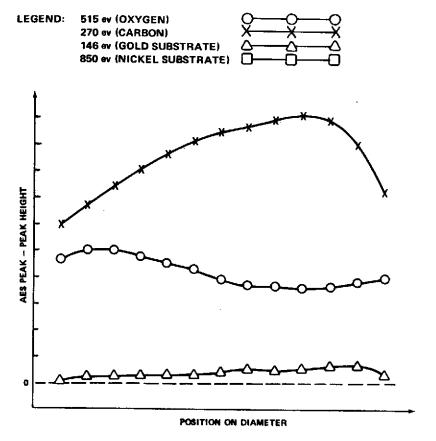
c. Sample no. 043K, -Y direction.

Figure 11. (Continued).



d. Sample no. 043M, +X direction.

Figure 11. (Concluded).



a. Sample no. 043N.

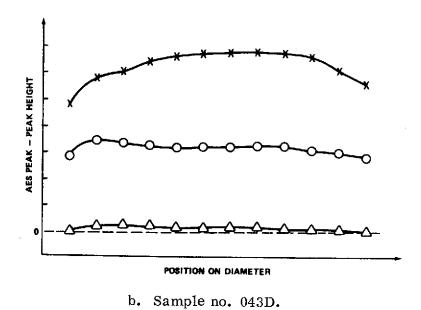
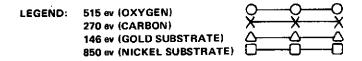


Figure 12. Postflight, gold control specimen in orbit.



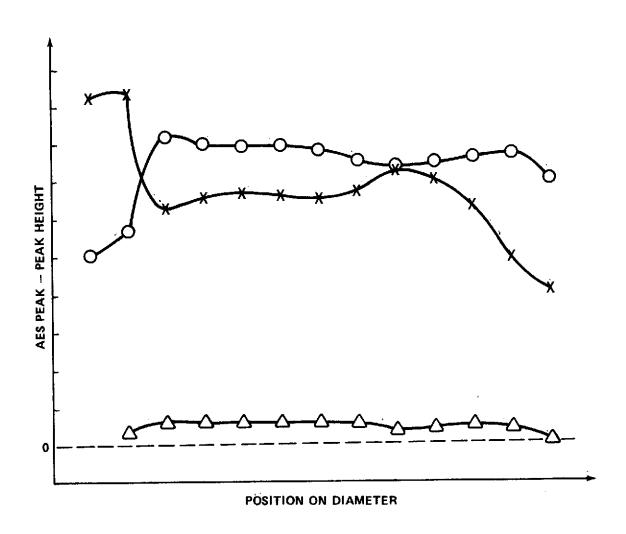
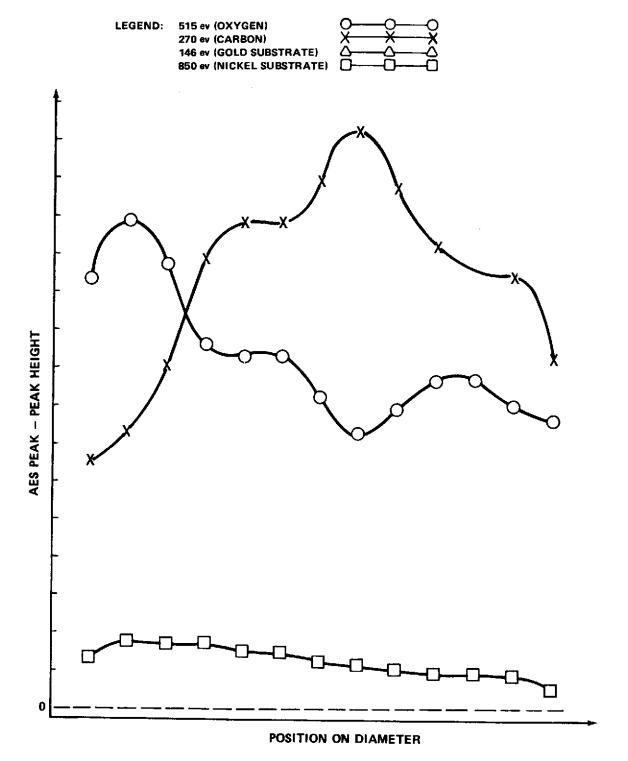
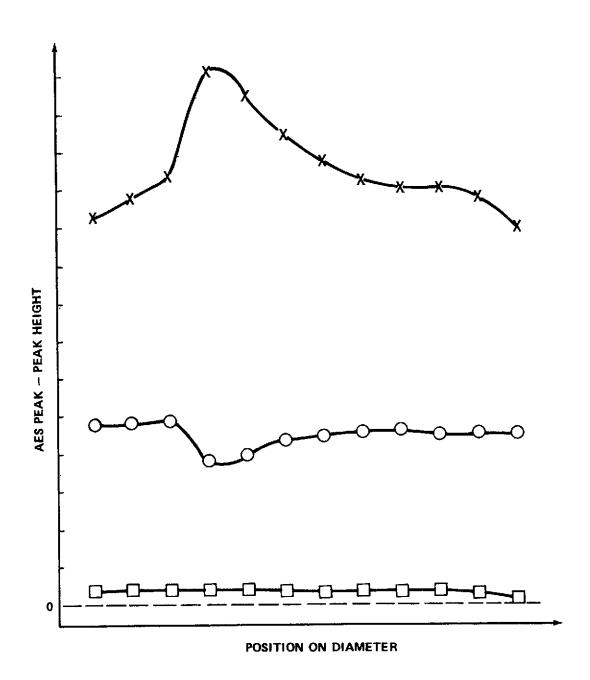


Figure 13. Postflight, gold ground control specimen.



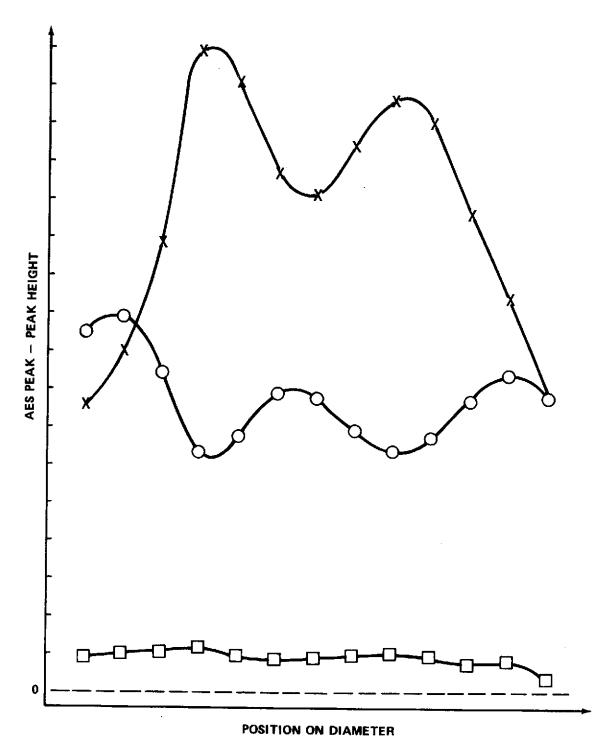
a. Sample no. 043H, -X direction.

Figure 14. Postflight, nickel specimen exposed in four directions.



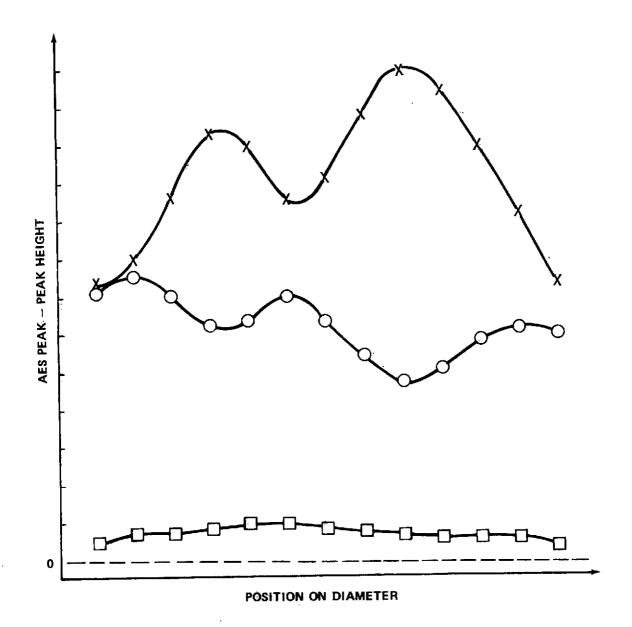
b. Sample no. 043E, +Y direction.

Figure 14. (Continued).



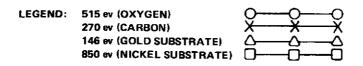
c. Sample no. 043K, -Y direction.

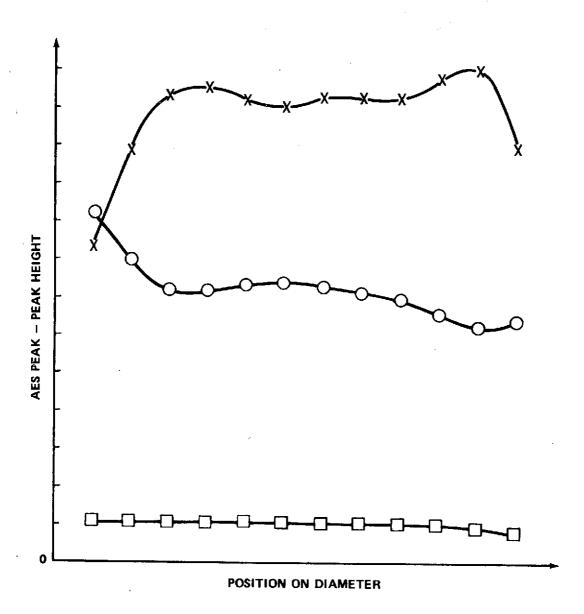
Figure 14. (Continued).



d. Sample no. 043M, +X direction.

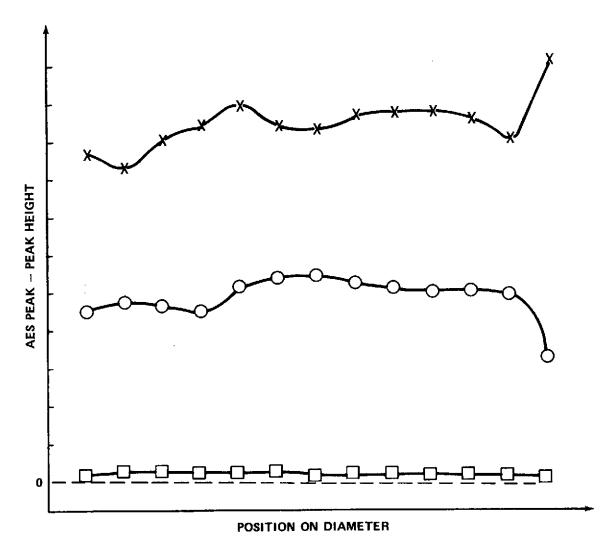
Figure 14. (Concluded).





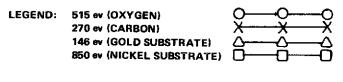
a. Sample no. 043N.

Figure 15. Postflight, nickel control specimen in orbit.



b. Sample no. 043D.

Figure 15. (Concluded).



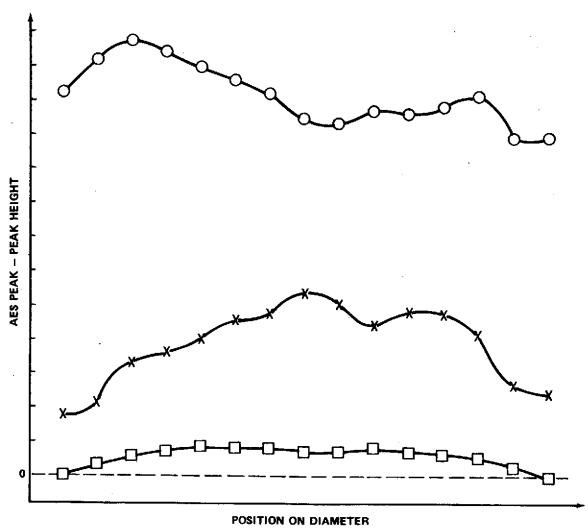
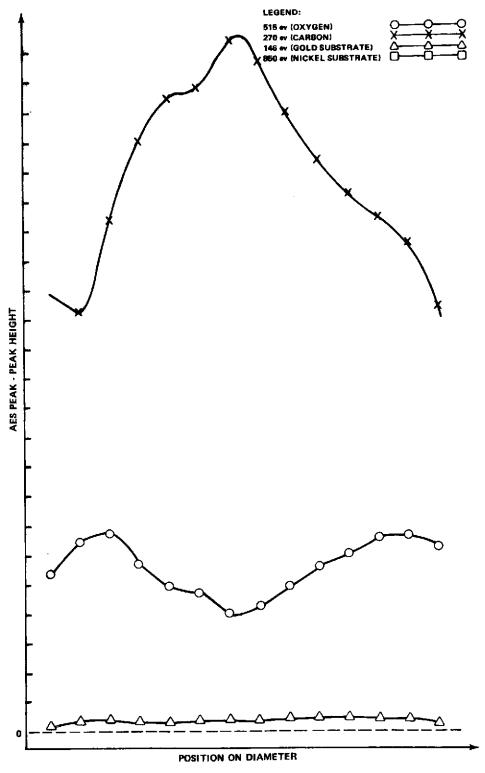
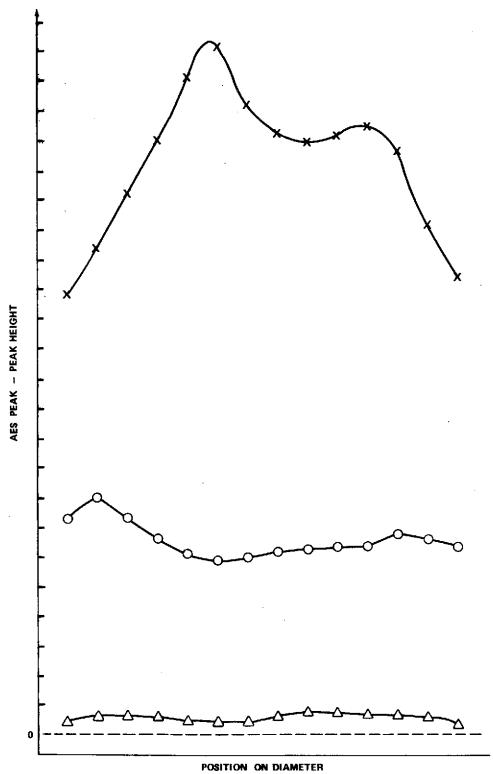


Figure 16. Postflight, nickel ground control specimen.



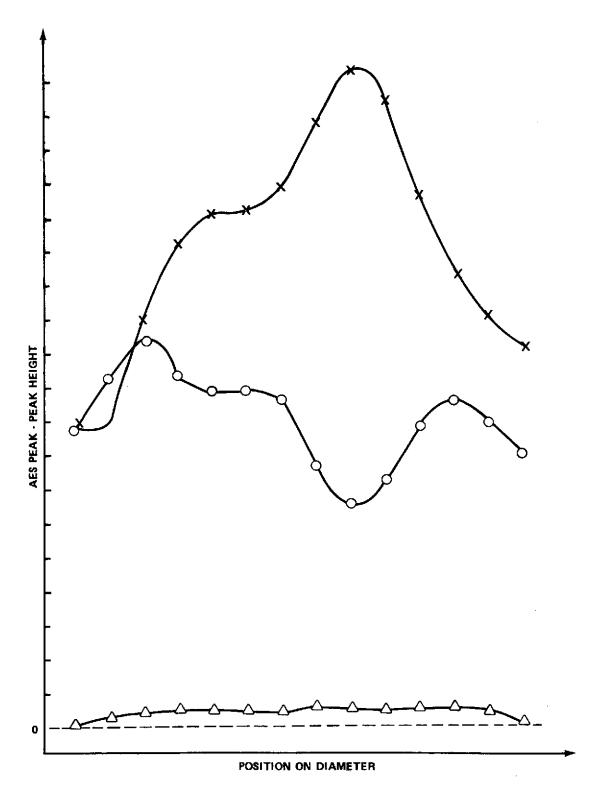
a. Sample no. 043H.

Figure 17. Postflight, gold specimen after 7 weeks in vacuo.



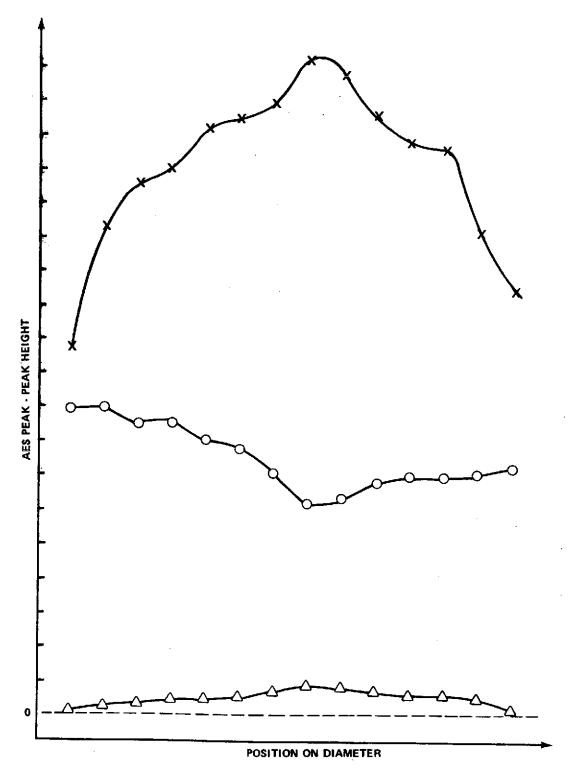
b. Sample no. 043E.

Figure 17. (Continued).



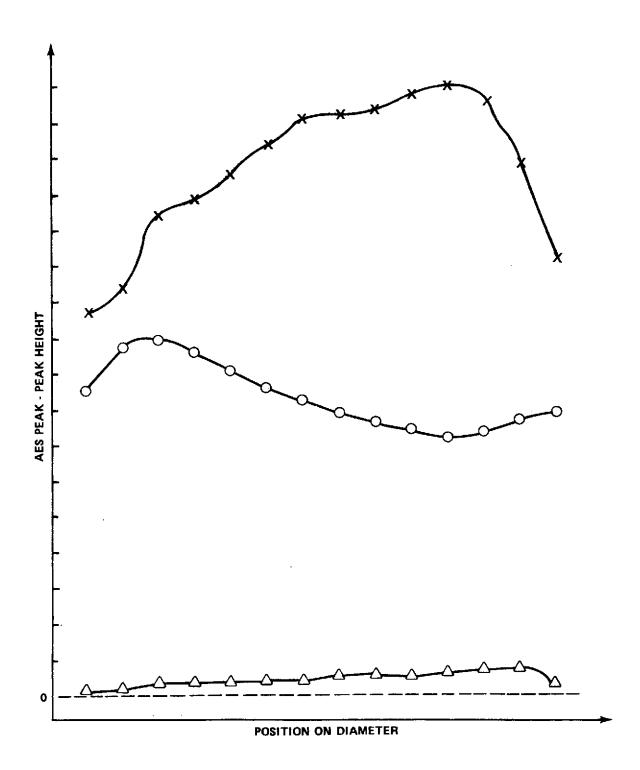
c. Sample no. 043K.

Figure 17. (Continued).



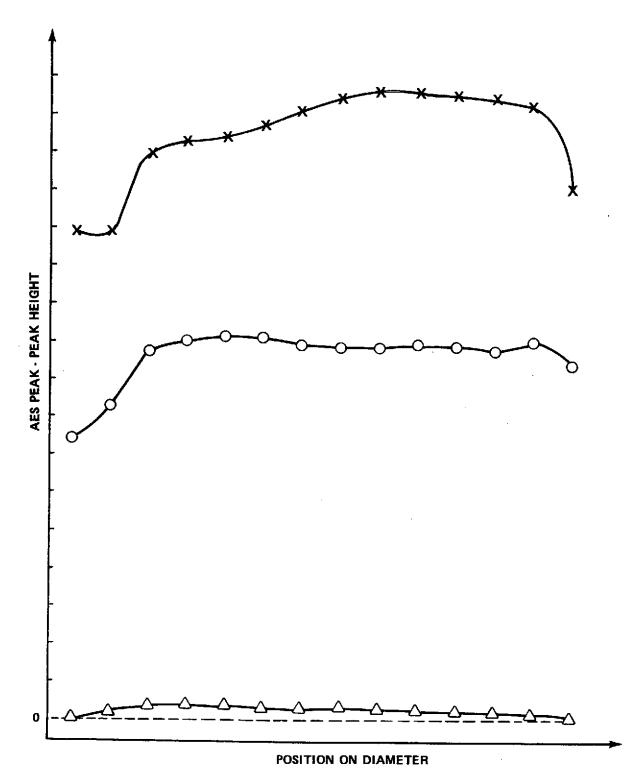
d. Sample no. 043M.

Figure 17. (Continued).



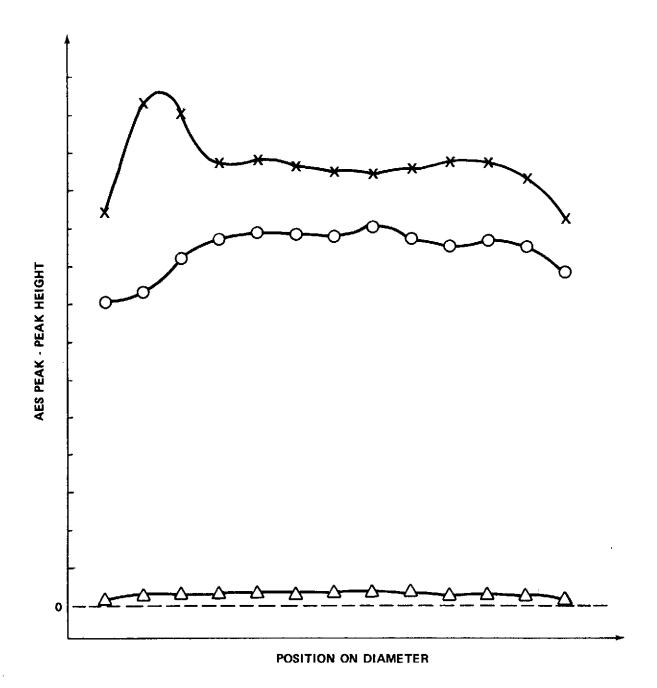
e. Sample no. 043N.

Figure 17. (Continued).



f. Sample no. 043D.

Figure 17. (Continued).



g. Sample no. 043J.

Figure 17. (Concluded).

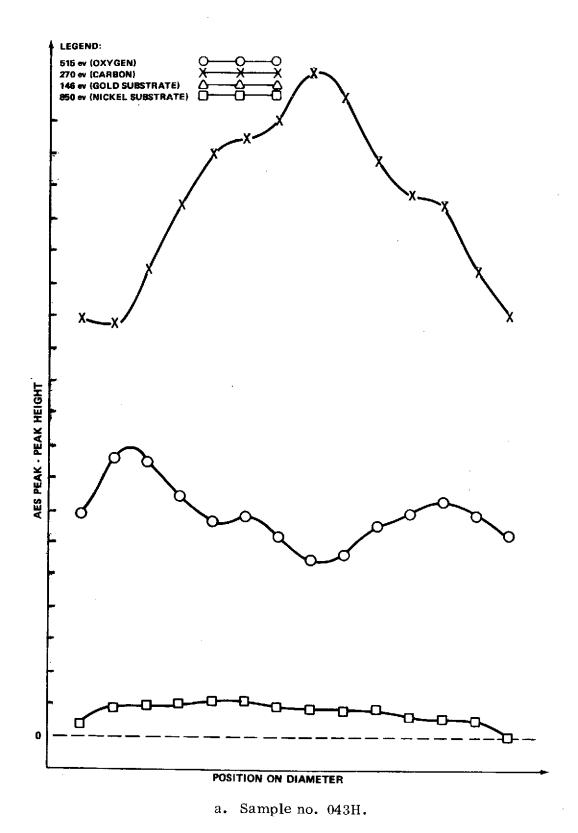
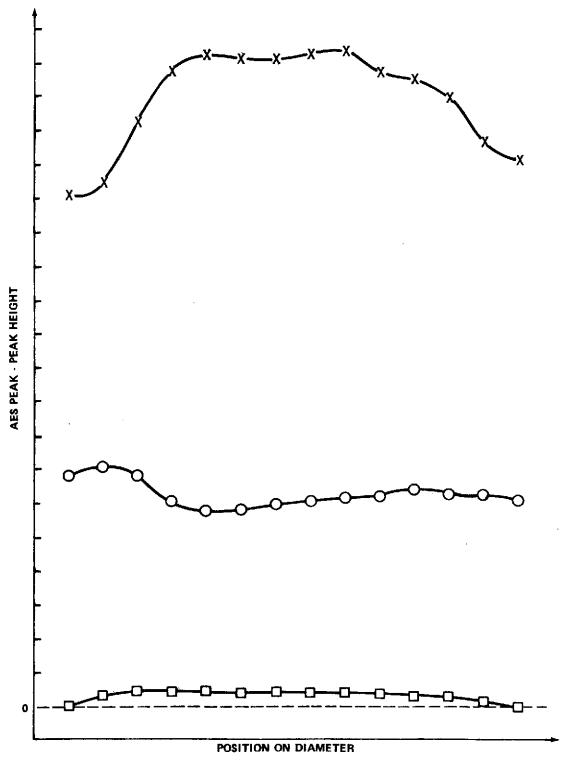
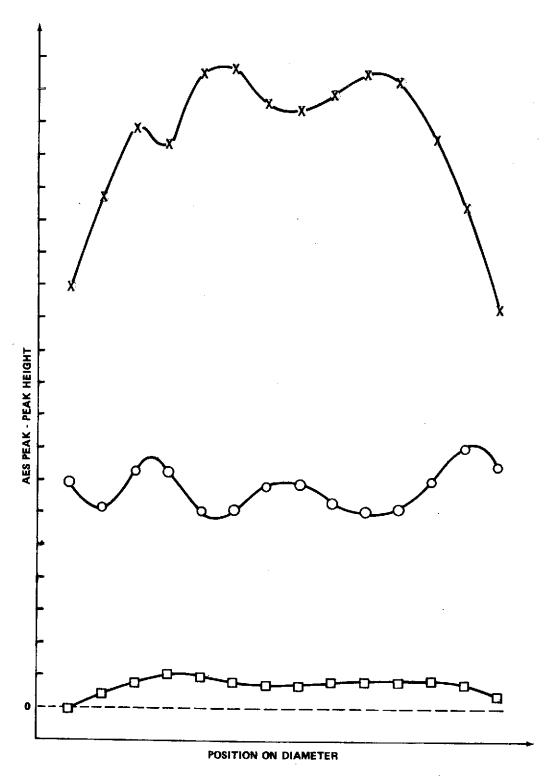


Figure 18. Postflight, nickel specimen after 7 weeks in vacuo.



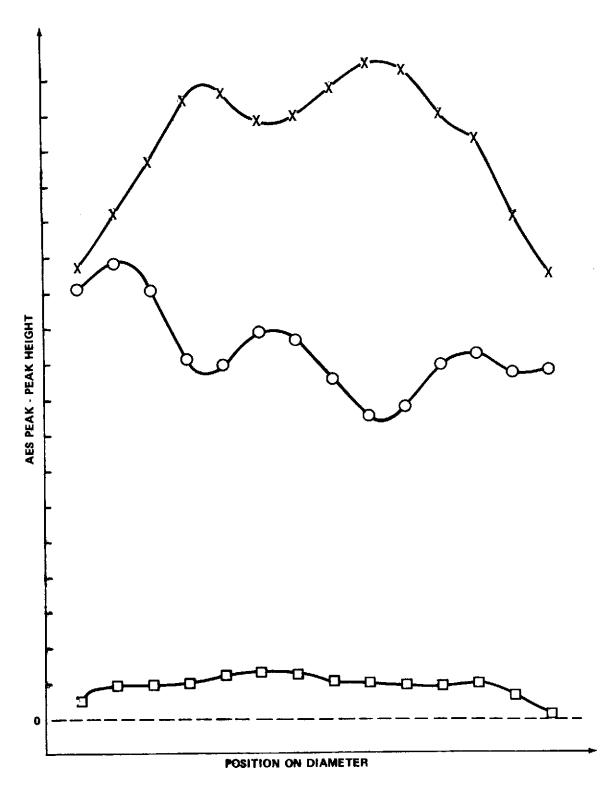
b. Sample no. 043E.

Figure 18. (Continued).



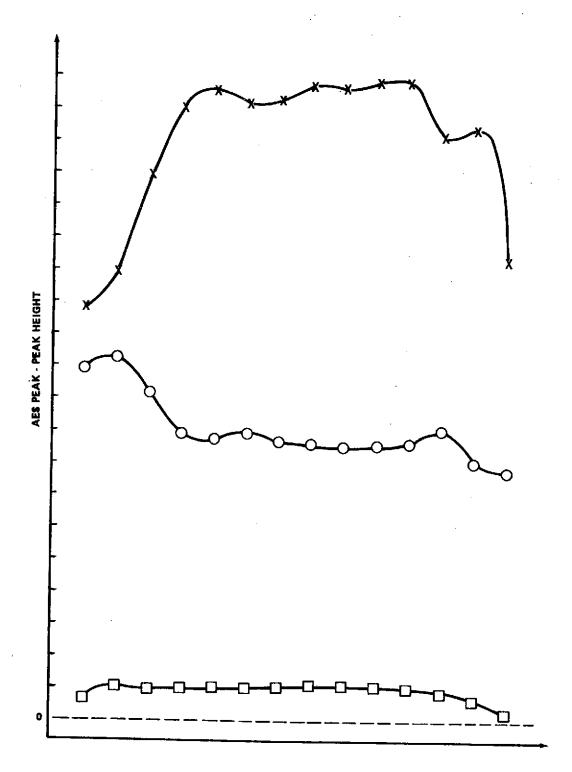
c. Sample no. 043K.

Figure 18. (Continued).



d. Sample no. 043M.

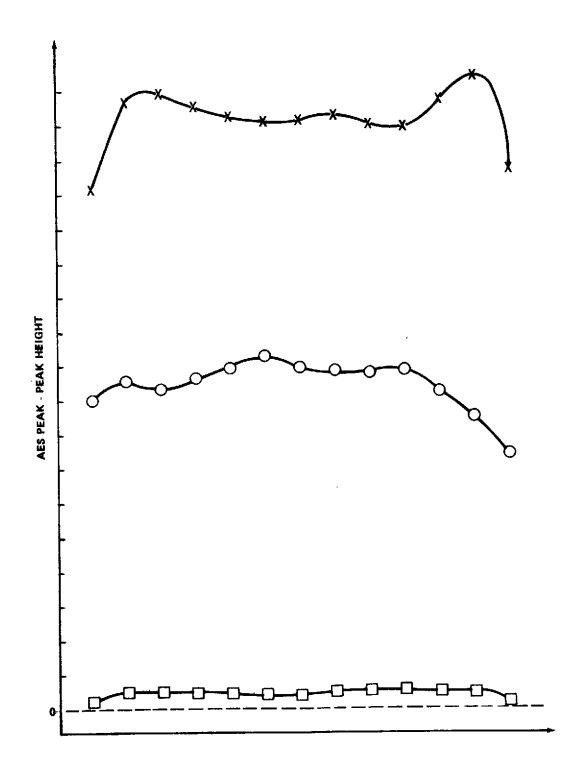
Figure 18. (Continued).



POSITION ON DIAMETER

e. Sample no. 043N.

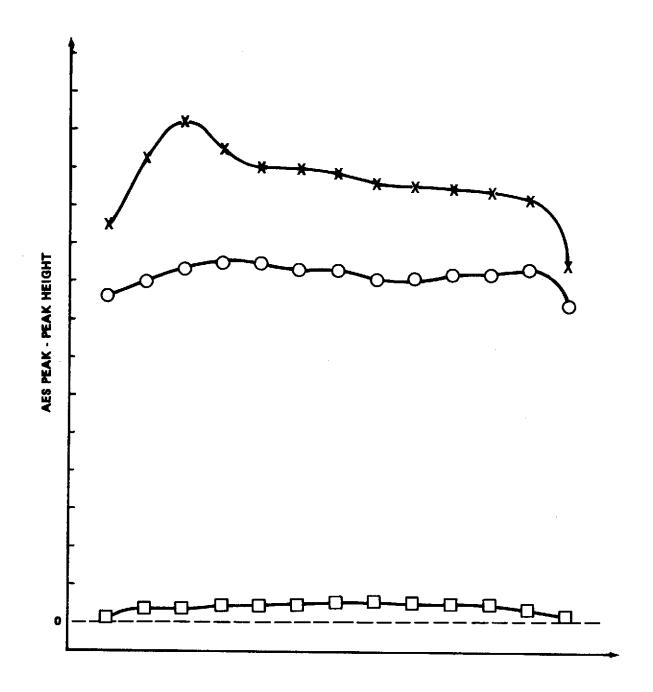
Figure 18. (Continued).



POSITION ON DIAMETER

f. Sample no. 043D.

Figure 18. (Continued).



POSITION ON DIAMETER

g. Sample no. 043J.

Figure 18. (Concluded).

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APPROVAL

AUGER MEASUREMENTS ON T-027 SAMPLES EXPOSED DURING THE SKYLAB 2 MISSION

By Palmer N. Peters

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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